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AMENDED SPECIFICATION

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PATENT SPECIFICATION



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435,345

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Accepted: Sept. 19, 1935.

PROVISIONAL SPECIFICATION

Improvements in or relating to the Manufacture of Activated Carbon

I, ROBIN GEORGE WESTBURY FARNELL, F.I.C., a British Subject, of Idleigh Court, Meopham, Kent, do hereby declare the nature of this invention to be as follows:—

This invention is for improvements in or relating to the manufacture of activated carbon and deals particularly with the manufacture of an activated carbon of low apparent density and of high activity. It has been found that the black ash residue obtained as a by-product from the digestion of esparto grass with alkali constitutes a material—particularly suitable for the manufacture of an activated carbon having the desirable properties mentioned above.

The present invention accordingly comprises a process for the preparation of activated carbon from the black ash residue resulting from the digestion of materials containing pecto-cellulose (for example esparto grass) with alkali which comprises heating the black ash in the presence of limited amounts of oxygen to a temperature between 700 and 1000° C. and thereafter continuing the heating in the presence of an excess of oxygen at a temperature between 400 and 500° C.

In a preferred form of the invention the black ash residue before being employed in the present process is leached with water until it is substantially free from alkali, for example until it has a pH between 9 and 10.

The black ash is preferably carbonised in a thin layer and is continually moved so as to expose fresh surfaces. This stage of the process may conveniently be carried out in a Herreschoff oil-fired furnace, the proportion of oxygen in contact with the black ash being controlled by the dilution of air admitted to the furnace with dry steam. The temperature to which the carbonised black ash is raised at this stage of the process depends on the purpose for

[Price 1/-]

which the activated carbon is required, active carbons of the highest activation being obtained by treatment at the highest temperature. For certain purposes, for example the decolorisation of vegetable oil and the removal of taste and odour from water, the carbonised black ash leaving the furnace may be cooled and ground, further activating treatment being omitted. In order to obtain a carbon of greater activity however the product leaving the furnace is subjected to the second treatment at a lower temperature. The apparatus employed for this second treatment consists of a horizontal hot plate within a furnace casing maintained at a temperature between 400° and 500° C. The temperature is maintained by admission of excess of air.

Conveniently the carbon leaving the furnace in which preliminary heat treatment takes place is fed directly to the second furnace.

When a neutral or an acid carbon is required limited amounts of mineral acid, for example 4N to 6N hydrochloric, phosphoric or sulphuric acid may be sprayed upon the hot carbon immediately before it leaves the second furnace.

When it is desired to reduce the ash content of the activated carbon it may be washed with water or with dilute mineral acid and dried. In this way the ash content may be reduced from say 15% to 3%.

When activated carbon of increased density is required there may be added to the black ash residue containing pecto-cellulose a proportion of wood, cocoanut or other ligno-cellulosic material.

Following is a description by way of example of one way of carrying the invention into effect employing as raw material the black ash residue obtained by evaporating to dryness the liquor obtained by digesting esparto grass with alkali in the manufacture of esparto paper. The black ash

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residue is washed substantially free from alkali by leaching with water for 12 to 36 hours. The washed ash is fed mechanically to an oil fired Herreschoff furnace 5 having four or more super-imposed circular hearths each provided with rabble arms which move the black ash from the centre to the periphery and from the periphery to the centre in successive hearths. 10 The products of combustion of the furnace sweep over the hearths and the temperature increases from hearth to hearth until it reaches a temperature of 900° C. at the outlet of the last hearth. Dry steam is admitted to the bottom hearth of the furnace 15 to control the degree of oxidation of the carbon. Carbon leaving this furnace is fed at a temperature from 400 to 500° C. to a hot plate maintained at this temperature and provided with rabble arms 20 for moving the carbon in a spiral path from the inlet at the centre of the hot plate to the discharge at the periphery of

the hot plate. The hot plate is enclosed in a housing to which is admitted an 25 excess of oxygen sufficient to maintain the temperature of the hot plate by oxidation. Immediately before the carbon leaves the hot plate it is sprayed with a 5N solution of hydrochloric acid in order to neutralise 80 traces of alkali.

On leaving the hot plate the activated carbon is cooled and ground for example in a ball mill until 70% passes 200 mesh (I.M.M.) and 90% passes 100 mesh 85 (I.M.M.)

The activated carbon thus obtained has a very low apparent density (about 10 to 15 pounds per cubic foot) and possesses 40 great activity.

Dated this 1st day of September, 1934.  
BOULT, WADE & TENNANT,  
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Chartered Patent Agents.

#### COMPLETE SPECIFICATION

#### Improvements in or relating to the Manufacture of Activated Carbon

I, ROBIN GEORGE WESTBURY FARNELL, F.I.C., a British Subject, of Idleigh Court, Meopham, Kent, do hereby declare the nature of this invention and in what 45 manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention is for improvements in or relating to the manufacture of activated 50 carbon and deals particularly with the manufacture of an activated carbon of low apparent density and of high activity. It has now been found that the black ash residue resulting from the digestion of 55 esparto grass with alkali and containing a considerable proportion of pecto-cellulose is particularly suitable for the manufacture of an activated carbon having the desirable properties mentioned above.

60 The present invention accordingly consists in the process for the preparation of activated carbon which comprises leaching with water the black ash residue resulting from the digestion of esparto grass 65 with alkali until it has a pH between 9 and 10, heating the residue to a temperature between 700 and 1000° C. in the presence of limited amounts of air or oxygen and thereafter continuing the heating in 70 the presence of an excess of air or oxygen at a temperature between 400 and 500° C.

The black ash after being dried, if desired, e.g. in a shelf drier heated by flue gases from the heating furnaces, is preferably carbonised in a thin layer and is 75 continually moved so as to expose fresh

surfaces. Alternatively, the black ash may be fed to the furnace while still wet and containing up to 67% to 75% moisture. This stage of the process may conveniently be carried out in a Herreschoff oil-fired furnace, the proportion of oxygen in contact with the black ash being controlled by diluting the air admitted to the furnace with dry steam. The temperature to which the carbonised black ash is raised at this stage of the process depends on the purpose for which the activated carbon is required, active carbons of the highest activation being obtained by treatment at the highest temperature. A carbon of lower activity suitable for example for the decolorisation of vegetable oil and the removal of taste and odour from water may be obtained by omitting the second heat-treatment, but this forms no part of my invention. The apparatus employed for the second treatment consists of a horizontal hot plate within a furnace casing maintained at a temperature between 400° and 500° C. The temperature is maintained by combustion of part of the carbon undergoing activation and regulation of the temperature may be effected by controlling the amount of air supplied to the hot plate. In one form of apparatus for carrying out this step of the process the temperature may be raised by blowing air on to the carbon and lowered by shielding the carbon from excess of air by sliding screens placed around and over the hot plate.

Conveniently the carbon leaving the furnace in which preliminary heat treatment takes place is fed directly to the second furnace.

5 In one modification of the present invention the carbonised black ash after the first heating may be reheated in the same furnace (or a part of the same furnace) to the lower temperature above specified.

10 When a neutral or an acid carbon is required mineral acid, for example 4N to 6N hydrochloric, phosphoric or sulphuric acid may be sprayed upon the hot carbon immediately before it leaves the second

15 furnace. Thus when a neutral carbon is required six gallons of commercial hydrochloric acid diluted with about four times its volume of water is sprayed upon each ton of carbon. When an acid carbon is required eight gallons of commercial hydrochloric acid diluted with about four times its volume of water may be used per

20 ton of carbon.

When it is desired to reduce the ash content of the activated carbon it may be washed with water or with dilute mineral acid and dried. In this way the ash content may be reduced from say 15% to 3%.

25 In an alternative method of operation the carbon leaving the furnace in which preliminary heat takes place is treated with mineral acid before the second heating. This form of the process avoids the necessity for a special drying step after

30 treatment of the carbon with acid.

When activated carbon of increased density is required there may be added to the esparto grass or to the black ash residue of such material a proportion of 40 wood (in the form of chips or sawdust), wood charcoal, coconut shell or coconut shell charcoal, corozo nuts, apricot stones or other ligno-cellulosic material.

Following is a description by way of example of one way of carrying the invention into effect. The raw material employed is the black ash residue obtained by evaporating to dryness the liquor obtained by digesting esparto grass with 50 alkali in the manufacture of esparto paper. The black ash residue is washed substantially free from alkali by leaching with water for 12 to 36 hours. The washed ash is fed mechanically to an oil-fired 55 Herreschoff furnace having four or more superimposed circular hearths each provided with rabble arms which move the black ash from the centre to the periphery and from the periphery to the centre in successive hearths. The products of combustion of the furnace sweep over the hearths and the temperature increases from hearth to hearth until it reaches a temperature of 900° C. at the outlet of 60 the last hearth. Dry steam is admitted

to the bottom of the hearth of the furnace to control the degree of oxidation of the carbon. Carbon leaving this furnace is fed at a temperature from 400° to 500° C. to a hot plate maintained at this temperature and provided with rabble arms for moving the carbon in a spiral path from the inlet at the centre of the hot plate to the discharge at the periphery of the hot plate. The hot plate is enclosed in a housing to which is admitted an excess of oxygen sufficient to maintain the temperature of the hot plate by oxidation. Immediately before the carbon leaves the hot plate it is sprayed with a 5N solution of hydrochloric acid in order to neutralise traces of alkali.

70 On leaving the hot plate the activated carbon is cooled and ground for example in a ball mill until 70% passes 200 mesh (I.M.M.) and 90% passes 100 mesh (I.M.M.).

75 The activated carbon thus obtained has a very low apparent density (about 10 to 15 pounds per cubic foot) and possesses great activity. This property of low density is of particular advantage when the carbon is to be employed in the purification of water because the carbon remains in suspension for an increased 80 length of time.

85 One form of activated carbon suitable for the decolorisation of cotton seed oil may be produced from a mixture in equal weights of highly active esparto carbon 90 of low ash content (e.g. having an ash content not greater than 3%) and finely ground inactive content charcoal also having an ash content not exceeding 3%. It has been found that in certain cases 100 such as for example the removal of the red colour of cotton seed oil it is advantageous to mix a proportion (e.g. 25 to 50%) of the powdered animal charcoal known as Ivory Black with the esparto 105 carbon.

95 In Specification No. 300,146 it has been proposed to activate or revivify carbonaceous material obtained for example by the calcination of the dissolved vegetable matter contained in the waste liquors from the manufacture of paper pulp by the caustic soda process by first purifying the carbonised material by acid washing or similar treatment and thereafter heating in an electric furnace first to a temperature of 800 to 900° C. in the presence of limited amounts of air or oxygen and subsequently at a temperature between 115 350 and 500° C. in the presence of air or 120 other oxidising gases. This specification however does not refer to the carbonising of the black ash residue resulting from the digestion of esparto grass with alkali nor does it refer to a black ash residue 125 180

which has been leached with water until it has a pH between 9 and 10.

In the Specification No. 225,891 a process has been described and claimed for treating activated carbon with a limited quantity of an acid solution which is all retained in the carbon to yield a product containing all the natural ash.

Having now particularly described and ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:—

1. The process for the preparation of activated carbon which comprises leaching with water the black ash residue resulting from the digestion of esparto grass with alkali until it has a pH between 9 and 10, heating the residue to a temperature between 700 and 1000° C. in the presence of limited amounts of air or oxygen and thereafter continuing the heating in the presence of an excess of air or oxygen at a temperature between 400 and 500° C.

2. A process as claimed in Claim 1 wherein the black ash is predried after leaching and prior to its heat treatment.

3. A process as claimed in Claim 1 or Claim 2 wherein the black ash is carbonised in a thin layer and is continuously moved to expose fresh surfaces.

4. A process as claimed in any one of the preceding claims wherein the temperature of the first heating is regulated by the admission of dry steam to the heating furnace.

5. A process as claimed in any one of the preceding claims wherein a neutral or acid carbon is produced by adding to the hot carbon after its first heat-treatment or during or immediately before its discharge from its second heat-treatment

limited amounts of dilute mineral acid e.g. hydrochloric or phosphoric acid.

6. A process as claimed in any one of the preceding claims wherein the ash content of the activated carbon is reduced by washing the carbon with water and subsequently heating.

7. A process as claimed in any one of the preceding claims wherein the activated carbon is obtained from the black ash residue from esparto grass and wherein its density is increased by mixing with the black ash residue prior to heating a proportion of wood or cocoanut shell, wood charcoal or cocoanut charcoal.

8. A process as claimed in any one of the preceding claims wherein the first heat treatment of the leached black ash is carried out in a Herreschoff or like furnace.

9. A process for the preparation of activated carbon substantially as described in the specific example hereinbefore set forth.

10. Apparatus for use in carrying out the second heat-treatment of black ash residue as claimed in any one of the preceding claims and consisting of a hot plate capable of being maintained at a temperature between 400 and 500° C. and provided with rabble arms for moving the carbon in a spiral path from an inlet at the centre of the hot plate to a discharge at the periphery of the hot plate.

11. Activated carbon whenever prepared or produced by any one of the processes hereinbefore particularly described and ascertained.

Dated this 4th day of December, 1934.  
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